

¹H-NMR (d₆-DMSO, 200 MHz): δ =1.49 (s, 9H, C(CH₃)₃), 1.78 (bs, =C—CH₃), 3.44 (m, 2 H, NCH₂), 4.06 (m, 2 H, OCH₂), 5.61, 5.89 (2 m, in each case 1 H, vinylidene H), 7.62, 8.29 (2 s, in each case 1 H, aromatic H), 8.40 (bs, 3 H, COOH) ppm.

FAB-MS (glycerol-DMF): m/z =422 ([M+H]⁺), 420 ([M-H]⁻).

Example 5

Benzene-1,3-dicarboxylic acid-4(carboxylic acid N-tert.-butyl-N-methacryloyloxyethylamide) (XIII)

Benzene-1,4-dicarboxylic acid-5(carboxylic acid N-tert.-butyl-N-methacryloyloxyethylamide) (XIV)

A solution of 185.27 g (1.000 mol) of N-tert.-butyl-2-aminoethyl methacrylate in 250 ml of methylene chloride was added dropwise to a mixture of 192.13 g (1.000 mol) of benzenetricarboxylic acid anhydride, 500 ml of dry methylene chloride and 303.57 g (3.000 mol) of dry triethylamine at room temperature, while stirring. After the mixture had been stirred at 41° C. for 4 hours, it was poured into 3 l of water, the mixture was acidified with half-concentrated sulphuric acid and the organic phase was separated off. The aqueous phase was extracted with methylene chloride and, after drying and stabilisation with 350 mg of 2,6-di-tert.-butylcresol, the combined organic phases were concentrated to give 284.60 g (78% of theory) of a beige TM coloured solid of a mixture of the two isomers (XIII) and (XIV) of benzenetricarboxylic acid mono-(N-tert.-butyl-N-methacryloyloxyethylamide).

Melting point: 71° C.

IR (KBr): ν =3400–2400, 1700, 1610, 1370, 1283, 1161, 1040, 1010, 932, 752 cm⁻¹.

¹H-NMR(CDCl₃, 200 MHz): for XIII: δ =1.58 (bs, 9 H, C(CH₃)₃), 1.82 (bs, 3 H, =CCH₃), 3.32 (m, 2 H, NCH₂), 4.51 (m, 2 H, OCH₂), 5.49, 5.95 (2 m, in each case 1 H, vinylidene H), 7.1 (m, 2 H, COOH), 8.15, 8.75 (m, 3 H, aromatic H) ppm.

for XIV: δ =1.43 (s, 9 H, C(CH₃)₃), 1.8 (bs, 3 H, =CCH₃), 3.48 (m, 2 H, NCH₂), 4.1 (m, 2 H, OCH₂), 5.44, 6.03 (2 m, in each case 1 H, vinylidene H), 7.1 (m, 2 H, COOH), 8.0–8.6 (m, 3 H, aromatic H) ppm.

Examples 6 to 10

(Preparation of the Formulations for Use as an Adhesive)

The adhesives according to the invention are produced by intensive mixing of the constituents listed in the following examples.

Example 6

37.5 g of water
50.0 g of tetrahydrofuran
12.5 g of trimellitic acid N-tert.-butyl-N-methacryloyloxyethylamide (IX) according to Example 2
0.02 g of camphorquinone

Example 7

27.3 g of water
63.6 g of tetrahydrofuran
9.1 g of trimellitic acid N-tert.-butyl-N-methacryloyloxyethylamide anhydride (VIII) according to Example 1
0.02 g of camphorquinone

Example 8

40.0 g of water
48.0 g of tetrahydrofuran
5 8.4 g of trimellitic acid N-tert.-butyl-N-methacryloyloxyethylamide (IX) according to Example 2
3.6 g of trimellitic acid N-tert.-butyl-N-methacryloyloxyethylamide anhydride (VIII) according to Example 1
10 0.02 g of camphorquinone

Example 9

93 7 g of ethanol
6.3 g of pyromellitic acid di(N-tert.-butyl-N-methacryloyloxyethylamide (p- and m-isomers (X) and (XI) according to Example 3)
15 0.01 g of camphorquinone

Example 10

20 43.5 g of water
43.5 g of tetrahydrofuran
9.5 g of pyromellitic acid (N-tert.-butyl-N-methacryloyloxyethylamide) (XII) according to Example 4
3.5 g of pyromellitic acid di(N-tert.-butyl-N-methacryloyloxyethylamide (p- and m-isomers (X) and (XI) according to Example 3)
25 0.02 g of camphorquinone

Example 11 (Use Test, Bonding Strength)

30 The activity and suitability of the adhesives (Examples 6–10) is checked by determination of the shear bonding strength to dentine. Human teeth which have been kept in 1% strength chloramine solution for a maximum of three months after the extraction are used.
35 Before being used in the test, the teeth are kept in physiological saline solution for at least three and not more than ten days, after thorough washing under running water. On the day before their use in the bonding test, the teeth are embedded individually, lying on an approximal side, with epoxy resin (®LEKUTHERM X 20, hardener T 3) in cylindrical rubber moulds of 25 mm diameter and 12 mm height. The teeth are ground by wet grinding on SiC papers of grains 240, 320, 400 and finally 600 to the extent that an adequately large dentine surface close to the enamel is exposed for binding to a cylinder of plastic of 3.5 mm diameter. After rinsing with deionised water and drying in a stream of air, the conditioning solution (®GLUMA 1 Cleanser is applied with a cotton-wool pellet using a rubbing movement for
50 30 seconds and the teeth are rinsed with water and dried, before the adhesive is applied with a brush, left on the surface for 30 seconds and then dried thoroughly in a stream of compressed air. One drop of (®GLUMA 4 Sealer is then applied and blown into a thin layer with compressed air. The sample pretreated in this way is firmly clamped in a clamping device under a divisible Teflon mould having a cylindrical receptacle 3.5 mm wide and 1 mm in height. The cylindrical mould is then filled with the plastic filling material (®PEKAFILL
60 (U) using a syringe, and the filling material is covered with a strip which is impermeable to O₂ and activated for 60 seconds under the supported light discharge opening of a (®TRANSLUX CL (Kulzer) polymerisation lamp. The sample is then immediately removed from the holder. The Teflon mould is removed and the sample is kept in warm water at 23° C. for 15 minutes, until shearing stress is initiated, this being effected with the aid of a pressure piston parallel to and close to the